CHAPTER III

EXPERIMENTATION

3.1 The Apparatus

The experimental apparatus as shown in Figure 3.4 and
Figure 3.5 can be divided in 3 parts: mixing system; radiation
detection and recording system; and conductivity measurement and
recording system.

3.1.1 Mixing System

The mixing system used consists of the following:

Mixing tank : A 24 cm in diameter and 33 cm high flat
bottom cylindrical tank equipped with 4 detachable baffles arranged
as shown in Figure 3.1. The baffle were 2.4 cm wide and 30 cm
high. The material used in the construction of all parts was
stainless steel. For the conductivity measurement purpose, two
platinum electrode holders were installed at 2.5 cm and 21.5 cm
above the bottom of the tank on opposite sides as shown in
Figure 3.2. At the center of the bottom of the tank an electric
controlled valve was connected to minimize exposure of radiation when
draining radioactive tracer contaminated water.

Agitator   : A US made Stirr-Pak variable speed motor model
C-4550 equipped with a controller unit, 0.8 cm in diameter drive
shaft and interchangeable impeller at the other end of the shaft was

used. Three types of impeller as shown in Figure 3.3 were used, i.e. 6 bladed disc turbine, 6 bladed open turbine, and 6 bladed 45 degree pitch turbine.

3.1.2 Radiation Detection and Recording

Radiation detection and recording consist of the following :

Detector : A US made EG&G ORTEC model 266, 2 in.x2 in. NaI(Tl) scintillation crystal equipped with photomultiplier tube and shielded with lead cylinder with an opening of 10 mm diameter.

Multichannel Analyzer : A US made Canberra multichannel analyzer model 8100 equipped with serial printer.

3.1.3 Conductivity Measurement and Recording

Conductivity measurement and recording consists of the following :

Electrodes : Two 0.1 mm in diameter and 2 cm long platinum wires with 1 cm contacting length.

Conductimeter : A Swiss made Metrohm Herisau conductimeter type 518

Recorder : A US made Quantachrome recorder model SR-4 equipped with continuous recording paper.

3.1.4 Auxilliary

Tachometer : A US made Lutron, digital tachometer model DT-2232

Injector : 5 ml. lead shielded syringe.

3.2 <u>Materials Used</u>

The material used in this study were :

Water : De-ionized water to minimize influence of ion content in water especially in the experiment using conductivity method.

Tracer :   There are various tracers, both radioactive and chemical substances, can be used so it was necessary to comply with the following requirements :[18,19]

- It should mixed easily with water.

- It   should   cause   only   negligible   disturbance   or modification of the system.

- It   should   be   detectable and analyzed accurately at low concentration.

- It should only be present in the water at a negligible or constant concentration.

- It should not react with the water or any other substance in such a way as to affect the measurement.

- It should be cheap.

a. Radioactive tracer   : Tc-99m in the form of $NaTcO_4$ in 2% saline solution was selected for use as radioactive tracer because its properties fit the requirements and for the following reasons :

- Half-life : With half-life of 6.02 hour, it is long enough to allow time to tranfer from the chemical laboratory to the work site and to conduct the experiment for the whole day. On the other hand, it requires a short storage time before being released to the environment.

- Radiation : Tc-99m emits gamma rays with energy of 136 keV and although the energy is relatively low it can still penetrate the wall of the mixing tank and the shell of the detector. In safety aspects it therefore does not require thick and bulky shielding materials. The detection efficiency (photo peak) at this energy is higher than 80 % (for 2 in.*2 in. NaI(Tl) detector).

- Physicals and Chemicals : Tc-99m in the form of $NaTcO_4$ easily dissolve in water.

- Others : In addition, Tc-99m in that form is already used in medical applications and it has been prepared for that purpose by the Office of Atomic Energy for Peace.

The total activity of the tracer used in each experiment was about 10 to 20 mCi.

b. Chemical tracer : There are many chemical tracers that fit the above requirements as shown in Table 3.1. Analytical grade NaCl was selected because its properties fit the above requirements and for the following reasons:

- Its conductivity is proportional to its concentration over a wide range.

- It is commonly used in the laboratory and is relatively cheap.

The concentration of NaCl used was 30 % (wt/vol)

Table 3.1 Some properties of main chemical tracer. [18,19]

Chemical	Detection limit	Remark
$Na_2Cr_2O_7 \cdot 2H_2O$	$2*10^{-1}$ mg./l	with direct analysis
	$2*10^{-3}$ mg./l	after reconcentration
NaCl	1-10 mg./l	depending on original
		conductivity
Rhodamine B	$2*10^{-4}$ mg./l	
$(C_{28}H_{31}ClN_2O_3)$		
Fluorescein	$5*10^{-3}$ mg./l	
$(C_{20}H_{10}O_5Na_2)$		

Other chemical tracers are Rhodamine Wt, $NaNO_2$, $MgSO_4 \cdot 4H_2O$, sulfo-rhodamine G, LiCl, etc.

3.3 The Procedure

The object of this experiment was to obtain tracer response after injection of the tracer and then observe or test the quality of homogeneity of the tracer in order to determine required mixing time.

The procedure may be summarized into two parts, radioactive tracer method and conductivity method.

3.3.1 Radioactive Tracer Method

a. The calibration

In the detection of radiation it is necessary to calibrate the measuring system because the result changes with the surrounding and configuration of the system. Although in this study it is not necessary to know the absolute value, the calibration should still be conducted to ensure that the detection system is reliable and to verify the appropriate window opening of the detector for that gamma energy. This was done by simulating the detection system as close as the actual case.

In order to verify the appropriate window opening and system calibration, the mixing tank was mounted on a support and filled with water to a height of 24 cm. The lead shielded detector as shown in Figure 3.8 was placed perpendicular to the wall of the tank at level of 16 cm above the bottom of the tank. The multichannel analyzer set in pulse height analysis (PHA) mode and fully open window was turned on and the high voltage was gradually increased to the operating voltage of the detector. The agitator was mounted on a support and turned on. A 5 mCi of sodium pertecnitate was injected into the tank and mixed well for about 5 minutes. Then the radiation was counted for 20 seconds. After a few seconds the peak of radiation would appear on the screen of the mutichannel analyzer. While collecting the radiation, the window width of the multichannel analyzer was identified by adjusting the lower and the upper control knob to the beginning and the end of the peak. The spectrum of Tc-99m and window width setting are shown in Figure 3.9.

In order to calibrate the system, the multichannel analyzer was switched to mutichannel scaling mode and set to collection time interval of 0.5 seconds. Then the radiation was collected for at least 50 seconds (100 data would be obtained). The collected data were printed out. Another 10 mCi was injected and mixed well for 5 minutes. Then the same procedure was conducted to obtained 4 sets of calibration information.

b. The experiment

The mixing tank was mounted on a support and filled with de-ionized water to a height of 24 cm. The lead shielded detector was placed perpendicular to the wall of the tank at the desired level. The agitator was mounted on a support at the desired impeller level as shown in Figure 3.4. The agitator was turned on and adjusted to the desired speed. The multichannel analyzer set in mutichannel scaling mode and set at collection time interval of 0.5 second was turned on. The voltage supply to the detector was gradually increased to the operating voltage of the detector. Then the background radiation was collected for at least 50 seconds (100 data would be obtained). After the background radiation was obtained, about 10-20 mCi of sodium pertecnitate was injected using a lead shield syringe at half way between the drive shaft and the wall of the tank. The radiation counting of the tracer was carried on until there was no variation in the radiation counts. The counting of the radiation was allowed further for at least 100 seconds to ensure getting a homogeneous solution. After completion of the experiment, the collected data were printed out.

3.3.2 Conductivity Method

a. The calibration

The mixing tank with the installed platinum electrodes was mounted on a support and filled with de-ionized water to a height of 24 cm. The conductivity of de-ionized water was measured using a known cell constant conductivity electrode. Then conductivity was again measured using Pt electrodes and the conductivity reading was adjusted to the value reading from known cell constant by adjusting the cell constant control knob. The agitator was mounted on a support and turned on. A 0.5 ml. of 30 % sodium chloride was injected into the tank and mixed well for about 5 minutes. The conductivity of the solution was measured using both types of electrode. Another 0.5 ml. of 30 % sodium chloride solution was injected and the same procedure was done to obtained 4 sets of calibration data.

b. The experiment

The mixing tank with the installed platinum electrods was mounted on a support and filled with de-ionized water to a height of 24 cm. The electrodes were connected to conductimeter and recorder. The conductimeter and recorder set at chart speed of 30 cm/min. were turned on. The agitator was mounted on a support and adjusted to the desired agitator level as shown in Figure 3.5. The agitator was turned on and adjusted to the desired speed. Then the conductivity of de-ionized water was measured and recorded to get background level. A 1 ml. of 30 % sodium choride solution was injected at half way between the wall of the tank and

the drive shaft. The changing of the conductivity of the mixing solution was recorded until there was no variation of the conductivity of the mixing solution. The measurement and the recording was allowed further for at least 20 seconds to ensure getting homogeneous solution. The experiments were conducted in a controlled room temperature at 26 °C.

The interested parameters in this study were effect by the speed of agitator, type of agitator, configuration of agitator, and baffle on required mixing time. The range of parameters studied were the follows :

speeds of impeller   : 200, 300, and 400 rpm

types of impeller   : 6 bladed open turbine, 6 bladed disc turbine, and 6 bladed 45 degree pitch turbine

positions of impeller : at 8 cm and 12 cm above the bottom of the tank (1/3 and 1/2 of diameter of the tank respectively)

baffles   : with, and without baffles installation

For radioactive tracer method, 3 positions of the detector were conducted to obtain the variation of tracer response at different levels of the solution in the tank. Those levels were the following :

level of detection   : at 8 cm, 12 cm, and 16 cm above the
                          bottom of the tank (1/3, 1/2, and 2/3
                          of diameter of the tank respectively)

In order to study the effect of the baffles, the baffles were detached from the wall of the tank then the above procedure was repeated.

3.4 Additional Work

3.4.1 The Apparatus

Some additional work about the continuous stirred tank and tubular vessel were conducted. The continuous stirred tank consists of the same parts as mentioned in 3.1.1. The tubular vessels were 10 litres in volume at 3 different sizes of polyvinyl chloride pipe as the follows :

	nominal diameter (in.)	inside diameter (cm)	length (cm)
large pipe :	6	15.1	60.6
medium pipe :	4	10.3	130.3
small pipe :	3	6.8	298.9

The pipes are closed both ends by plexiglass flange with 1/2 in. pipe connector.

3.4.2 The Procedure

a. Continuous stirred tank

The tank was mounted on a support and the water supply pipe was connected to the tank at the top of the tank as an inlet pipe and the bottom of the tank as an outlet pipe as shown in Figure 3.6. The inlet pipe was drilled and sealed with a rubber strip for injection purposes. An inline flow meter was installed to the inlet pipe. The outlet pipe is connected to a storage tank to collect radioactive contaminated water. The lead shielded detector was placed perpendicular to the outlet pipe as close as posible to the exit of the tank. The agitator was mounted on a support at the desired agitator level. The outlet valve was turned off and the tank was continuously filled with water at the desired volumetric flow rate to a height of 24 cm. Then the outlet valve was turned on to keep the water level in the tank at 24 cm. After a steady state was reached, the agitator was turned on and adjusted at speed of 400 rpm. The multichannel analyzer set in mutiscaling mode and set at 0.5 second interval of collection time was turned on. The background radiation was collected for at least 50 seconds. Then 20-30 mCi of sodium pertecnitate was injected using a lead shielded syringe at the injection point. The radiation at the outlet pipe was continuously detected and recorded until the the detected radiation was close to the background level. The detection of radiation was allowed further for at least 100 seconds to ensure covering the tail of the tracer. After completion of the experiment, the collected data were printed out.

b. Tubular vessel

The tubular vessel was mounted horizontally on a support. The water supply pipe was connected to vessel at the inlet end of the vessel. The inlet pipe was drilled and sealed with a rubber strip for injection purposes. An inline flow meter was installed to the inlet pipe line. The outlet pipe was connected to a storage tank to collect the radioactive contaminated water. The lead shielded detector was placed perpendicular to the outlet pipe as close as posible to the exit of the vessel as shown in Figure 3.7. The outlet valve was partially turned off and the vessel was continuously filled up with water at the desired volumetric flow rate. Then the outlet valve was turned on to keep the volumetric flow rate of water at the desired value. The multichannel analyzer set in multichannelscaling (MCS) mode and set at 0.5 second interval of collection time was turned on. After a steady state was reached, the background radiation was collected for at least 50 seconds. Then 10-20 mCi of sodium pertecnitate was injected using a lead shielded syringe at the injection point. The radiation at the outlet pipe was continuously detected and recorded until the detected radiation was close to the background level. The collection of radiation was allowed further for at least 100 seconds to ensure covering the tail of the tracer. After completion of the experiment, the collected data were printed out.
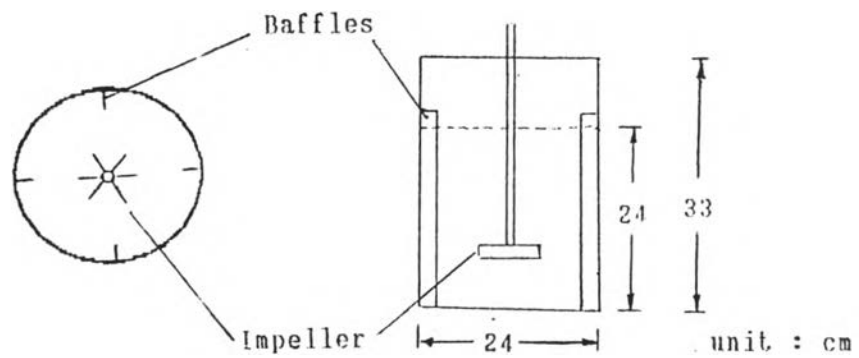
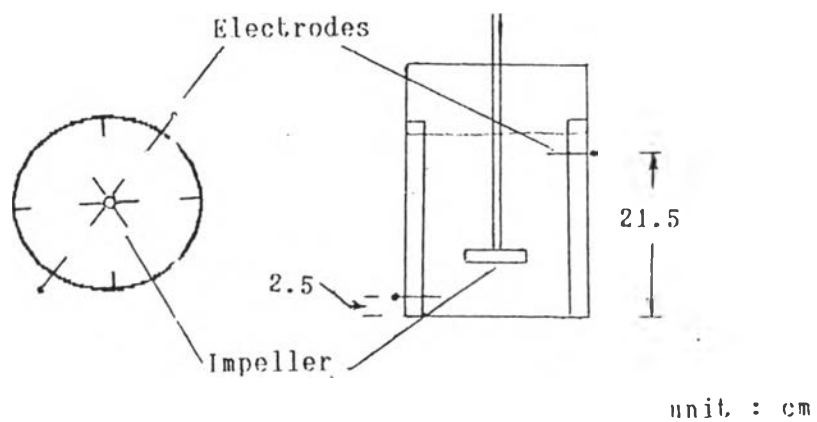Figure 3.1   Diagram of arrangement of impeller and baffles
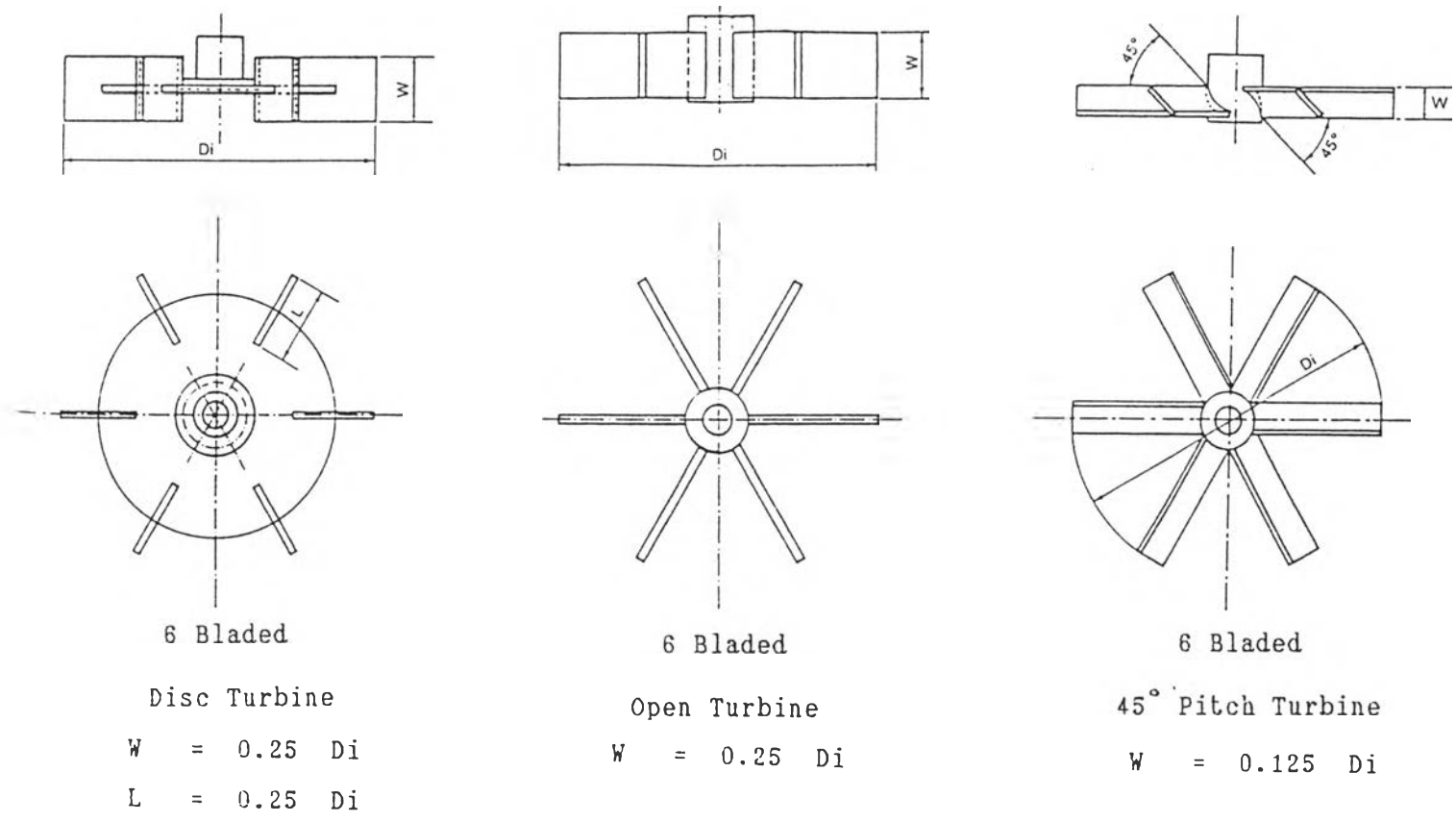

Figure 3.2   Diagram of electrode installation

6 Bladed

Disc Turbine

W = 0.25 Di

L = 0.25 Di

6 Bladed

Open Turbine

W = 0.25 Di

6 Bladed

45° Pitch Turbine

W = 0.125 Di
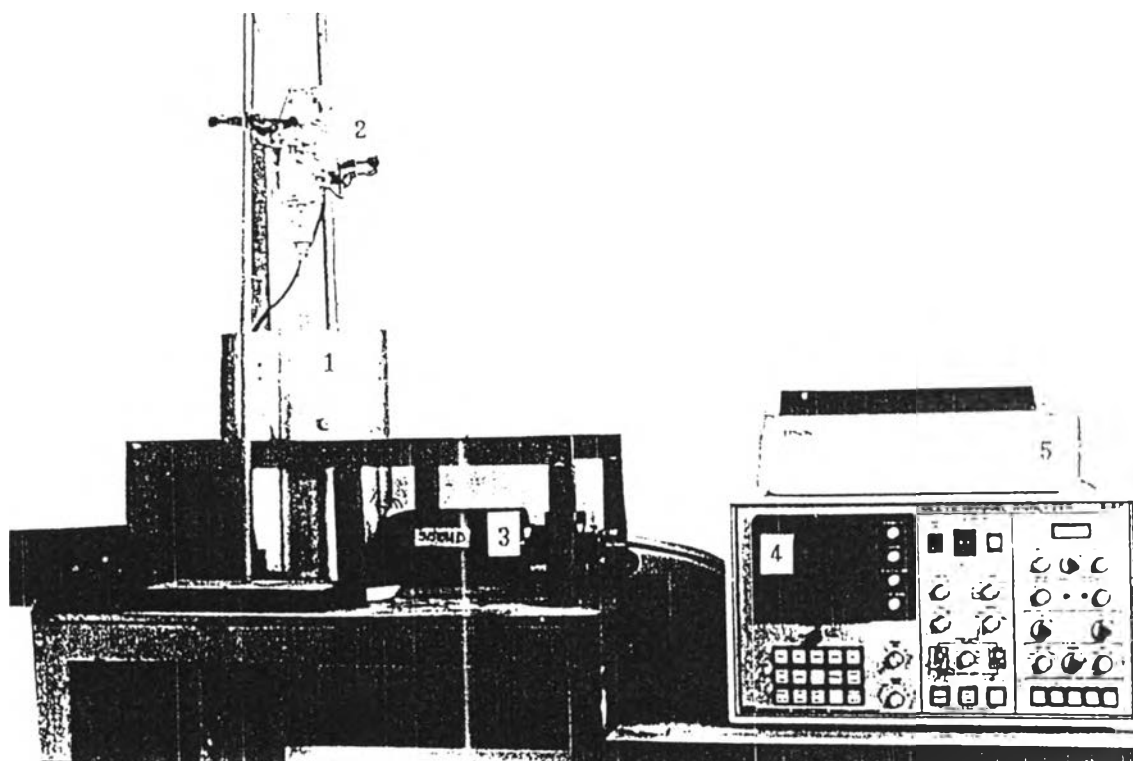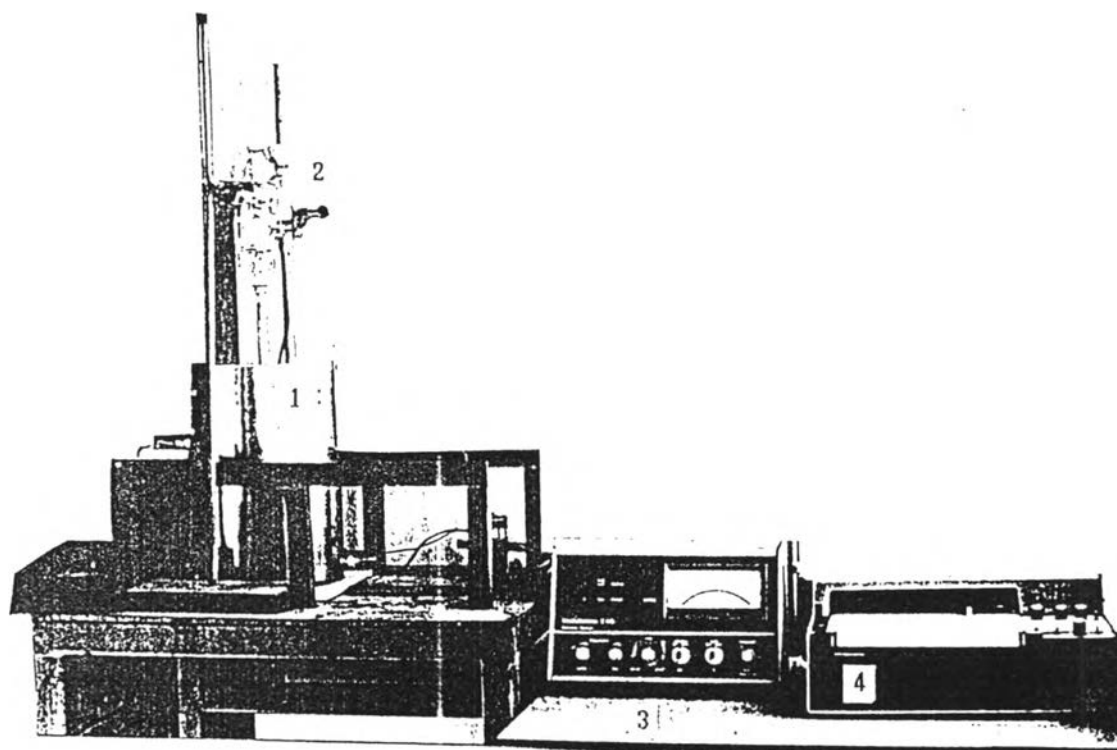
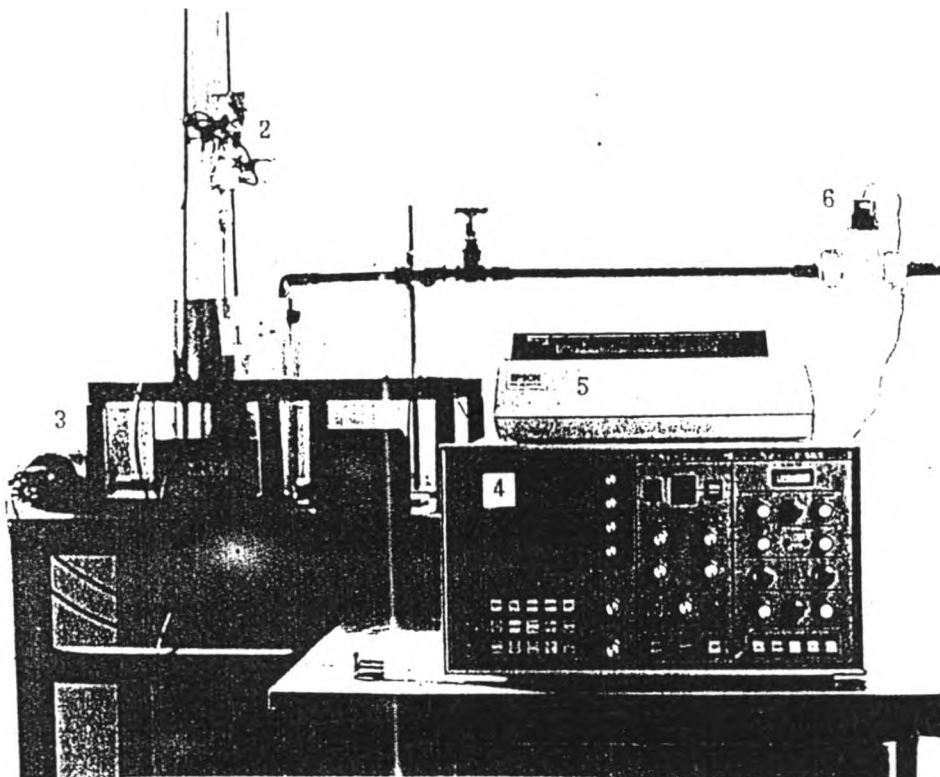Figure 3.3 Diagram of three types of considered impeller

Di = 8   cm

1. Mixing tank

2. Variable speed motor

3. Lead shielded detector

4. Multichannel analyzer

5. Printer

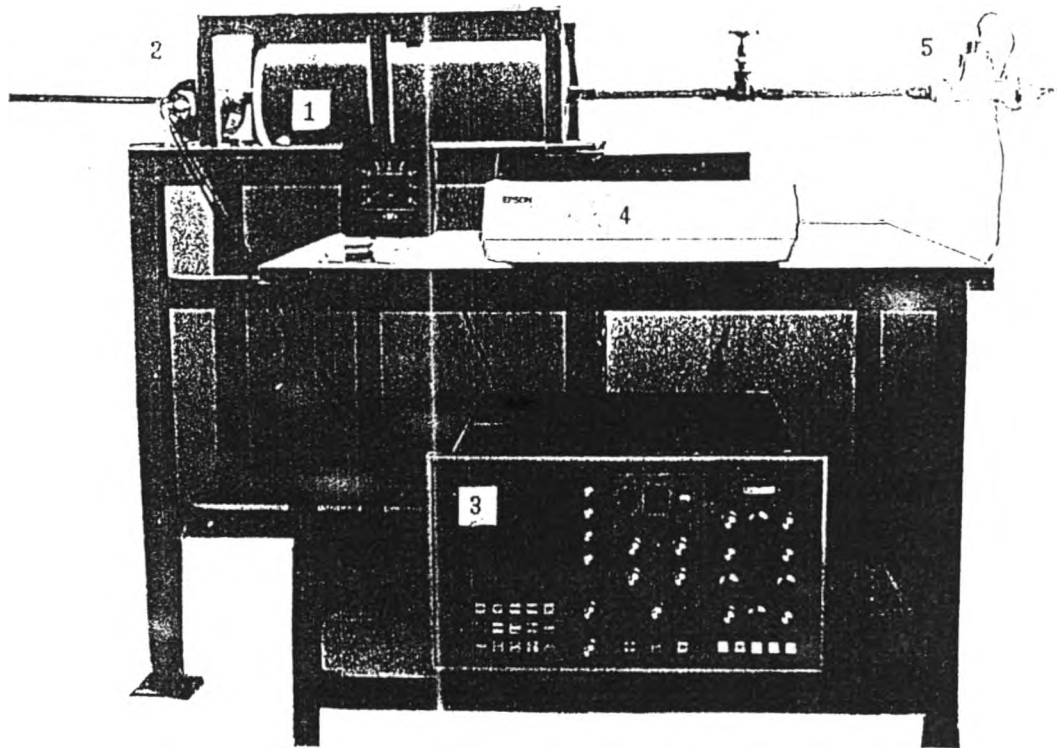Figure 3.4   The apparatus used in radioactive tracer technique

1. Mixing tank

2. Variable speed motor

3. Conductimeter

4. Chart recorder

Figure 3.5   The apparatus used in conductivity method

1 Continuous stirred tank

2 Variable speed motor

3 Lead shielded detector

4 Multichannel analyzer

5 Printer

6 Inline flow meter

Figure 3.6   The apparatus used in continuous stirred tank
using radioactive tracer technique

1 Tubular vessel

2 Lead shielded detector

3 Multichannel analyzer

4 Printer

5 Inline flow meter

Figure 3.7   The apparatus used in tubular vessel
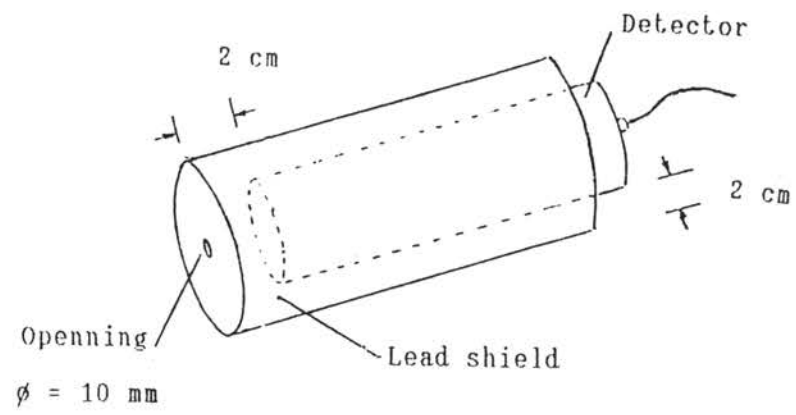using radioactive tracer technique
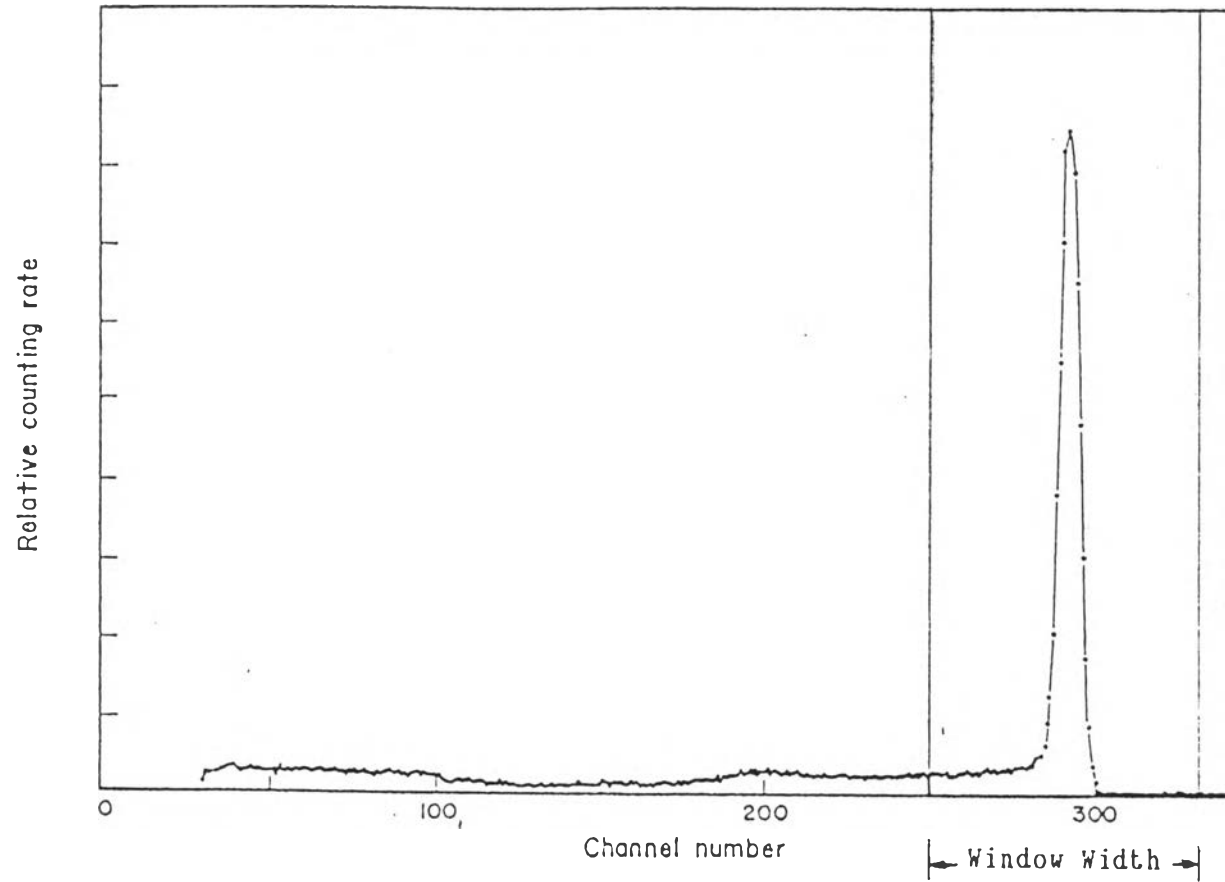
Figure 3.8 Diagram of lead shielded detector

Figure 3.9 Spectrum of ^{99m}Tc and setting of window width